

## 2,4-Dibromonaphthalen-1-ol

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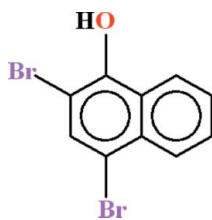
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.089; data-to-parameter ratio = 18.8.

In the essentially planar (r.m.s. deviation = 0.023 Å) title compound,  $\text{C}_{10}\text{H}_6\text{Br}_2\text{O}$ , an intramolecular  $\text{O}-\text{H}\cdots\text{Br}$  hydrogen bond generates an  $S(5)$  ring. In the crystal, molecules are linked by an  $\cdots\text{O}-\text{H}\cdots\text{O}-\text{H}\cdots\text{O}-\text{C}(2)$  chain extending along [100], which involves the same H atom that participates in the intramolecular hydrogen bond. Aromatic  $\pi-\pi$  interactions [centroid-centroid separation = 3.737 (4) Å] help to consolidate the packing.

## Related literature

For a related structure, see: Chanh *et al.* (1973); For graph-set notation, see: Bernstein *et al.* (1995).



## Experimental

## Crystal data

 $\text{C}_{10}\text{H}_6\text{Br}_2\text{O}$  $M_r = 301.97$ Orthorhombic,  $P2_12_12_1$  $a = 4.1225$  (3) Å $b = 14.4441$  (11) Å $c = 16.0490$  (14) Å $V = 955.65$  (13) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 8.44$  mm<sup>-1</sup> $T = 296$  K

0.32 × 0.14 × 0.12 mm

## Data collection

Bruker Kappa APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.254$ ,  $T_{\max} = 0.365$ 

5060 measured reflections

2239 independent reflections

1410 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.045$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$  $wR(F^2) = 0.089$  $S = 0.96$ 

2239 reflections

119 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.47$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.41$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

863 Friedel pairs

Flack parameter:  $-0.01$  (3)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{Br1}$	0.82	2.60	3.107 (5)	122
$\text{O1}-\text{H1}\cdots\text{O1}^i$	0.82	2.21	2.893 (6)	141

Symmetry code: (i)  $x - \frac{1}{2}, -y + \frac{1}{2}, -z$ .

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Ex-Vice Chancellor, University of Sargodha, Pakistan. ARR also acknowledges the Higher Education Commission, Government of Pakistan, for generous support of this research project (No. 20-819).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6335).

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**supplementary materials**

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## 2,4-Dibromonaphthalen-1-ol

A. R. Raza, A. Sultan and M. N. Tahir

### Comment

The crystal structure of 2-bromonaphthalene (Chanh, *et al.*, 1973) has been published which is related to the title compound (Fig. 1).

The molecule of the title compound is planar with r.m.s. deviation of 0.0234 Å. The Br2 atom has maximum deviation from the mean plane and its value is 0.0574 (27) Å. There exists an intra-molecular hydrogen bond of O—H···Br type (Table 1, Fig. 1) and complete S(5) ring motif (Bernstein *et al.*, 1995). The molecules are stabilized in the form of polymeric chains due to intermolecular H-bonding of O—H···O type (Table 1, Fig. 2). Due to these hydrogen bonds a chain of ···O—H···O—H···O— exists. The  $\pi$ - $\pi$  interactions between the benzene rings (C1—C6) and (C1/C6—C10) of the naphthalen group at a distance of 3.737 (4) Å help to consolidate the packing.

### Experimental

Bromine (2.9 ml, 9.2 g, 30 mmol, 2 eq) was added as drops to an ice-chilled solution of  $\alpha,\beta$ -unsaturated-1-tetralone (2.2 g, 15 mmol, 1 eq) in  $\text{CHCl}_3$  (50 ml) and was stirred for 1 h.  $\text{Et}_3\text{N}$  (3 ml, 2.2 g, 22 mmol, 1.5 eq) was added to the reaction mixture followed by 2 h stirring at room temperature. After the commencement of reaction, the reaction mixture was neutralized with aq HCl (15 ml). The organic layer was washed with  $\text{H}_2\text{O}$  ( $3 \times 25$  ml), dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure to afford the colorless needles of (I).

Yield: 2.4 g, 52%, m.p. 499 K.

### Refinement

The H-atoms were positioned geometrically with (O—H = 0.82, C—H = 0.93 Å) and refined as riding with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{O})$ , where  $x = 1.5$  for hydroxy and  $x = 1.2$  for aryl H-atoms.

### Figures

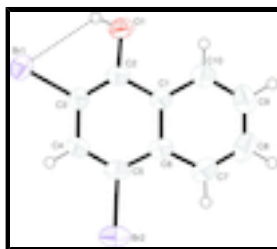


Fig. 1. View of the title compound with displacement ellipsoids drawn at the 50% probability level. The dotted line indicates the intramolecular H-bond.

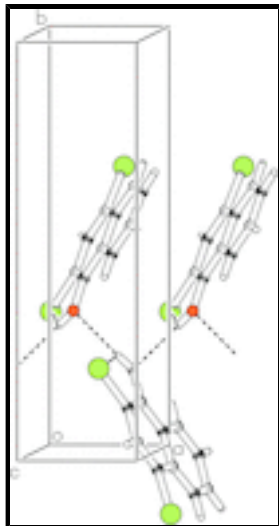


Fig. 2. The partial packing of (I), which shows that molecules form polymeric chains.

## 2,4-Dibromonaphthalen-1-ol

### Crystal data

$C_{10}H_6Br_2O$

$M_r = 301.97$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.1225$  (3) Å

$b = 14.4441$  (11) Å

$c = 16.0490$  (14) Å

$V = 955.65$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 576$

$D_x = 2.099$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1410 reflections

$\theta = 2.8$ – $27.9^\circ$

$\mu = 8.44$  mm<sup>-1</sup>

$T = 296$  K

Needle, colorless

$0.32 \times 0.14 \times 0.12$  mm

### Data collection

Bruker Kappa APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

Detector resolution: 7.60 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2005)

$T_{\min} = 0.254$ ,  $T_{\max} = 0.365$

5060 measured reflections

2239 independent reflections

1410 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 27.9^\circ$ ,  $\theta_{\min} = 2.8^\circ$

$h = -5 \rightarrow 5$

$k = -15 \rightarrow 19$

$l = -20 \rightarrow 20$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring  
sites

$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2]$
$S = 0.96$	where $P = (F_o^2 + 2F_c^2)/3$
2239 reflections	$(\Delta/\sigma)_{\max} < 0.001$
119 parameters	$\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.41 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 863 Friedel pairs
	Flack parameter: $-0.01 (3)$

*Special details*

**Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.45967 (15)	0.18900 (4)	0.18041 (4)	0.0493 (2)
Br2	1.06884 (18)	-0.15734 (4)	0.19773 (5)	0.0624 (3)
O1	0.6998 (11)	0.1798 (3)	-0.0029 (3)	0.0463 (16)
C1	0.9478 (14)	0.0326 (3)	0.0024 (3)	0.0333 (17)
C2	0.7730 (14)	0.1034 (4)	0.0449 (4)	0.0343 (19)
C3	0.6882 (14)	0.0934 (4)	0.1244 (4)	0.036 (2)
C4	0.7718 (13)	0.0149 (4)	0.1698 (4)	0.039 (2)
C5	0.9399 (15)	-0.0536 (4)	0.1318 (4)	0.0417 (19)
C6	1.0379 (14)	-0.0490 (4)	0.0474 (3)	0.0377 (19)
C7	1.2132 (16)	-0.1172 (4)	0.0024 (5)	0.048 (3)
C8	1.2958 (16)	-0.1080 (5)	-0.0777 (4)	0.058 (3)
C9	1.2149 (16)	-0.0287 (5)	-0.1202 (5)	0.059 (3)
C10	1.0428 (15)	0.0408 (4)	-0.0816 (3)	0.045 (2)
H1	0.57717	0.21391	0.02304	0.0692*
H4	0.71293	0.00947	0.22552	0.0471*
H7	1.27375	-0.17112	0.03010	0.0583*
H8	1.40791	-0.15515	-0.10460	0.0694*
H9	1.27695	-0.02190	-0.17559	0.0702*
H10	0.98850	0.09384	-0.11132	0.0537*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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## supplementary materials

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Br1	0.0513 (4)	0.0432 (3)	0.0535 (4)	-0.0002 (3)	0.0022 (4)	-0.0087 (3)
Br2	0.0679 (5)	0.0465 (4)	0.0727 (5)	0.0007 (3)	-0.0084 (4)	0.0200 (4)
O1	0.049 (3)	0.034 (2)	0.056 (3)	0.002 (2)	0.002 (2)	0.007 (2)
C1	0.030 (3)	0.036 (3)	0.034 (3)	-0.005 (3)	-0.008 (3)	-0.005 (3)
C2	0.034 (3)	0.030 (3)	0.039 (4)	-0.008 (3)	-0.009 (3)	0.003 (3)
C3	0.033 (3)	0.036 (4)	0.038 (4)	-0.008 (3)	0.000 (3)	-0.006 (3)
C4	0.041 (4)	0.039 (3)	0.038 (4)	-0.008 (3)	-0.006 (3)	0.005 (3)
C5	0.039 (3)	0.037 (3)	0.049 (4)	-0.006 (3)	-0.014 (3)	0.008 (3)
C6	0.033 (3)	0.031 (3)	0.049 (4)	-0.007 (3)	-0.012 (3)	-0.002 (3)
C7	0.047 (4)	0.042 (4)	0.056 (5)	0.004 (3)	-0.017 (4)	-0.012 (4)
C8	0.052 (4)	0.063 (5)	0.059 (6)	0.011 (4)	-0.004 (4)	-0.026 (5)
C9	0.052 (5)	0.081 (6)	0.043 (5)	-0.001 (4)	0.003 (4)	-0.006 (4)
C10	0.042 (4)	0.049 (3)	0.044 (4)	-0.002 (3)	-0.012 (3)	0.001 (3)

### Geometric parameters (Å, °)

Br1—C3	1.898 (6)	C5—C6	1.415 (8)
Br2—C5	1.910 (6)	C6—C7	1.419 (9)
O1—C2	1.377 (7)	C7—C8	1.337 (10)
O1—H1	0.8200	C8—C9	1.374 (10)
C1—C2	1.425 (8)	C9—C10	1.377 (9)
C1—C10	1.409 (7)	C4—H4	0.9300
C1—C6	1.431 (7)	C7—H7	0.9300
C2—C3	1.331 (9)	C8—H8	0.9300
C3—C4	1.391 (8)	C9—H9	0.9300
C4—C5	1.353 (8)	C10—H10	0.9300
C2—O1—H1	110.00	C1—C6—C5	116.6 (5)
C2—C1—C6	118.7 (5)	C6—C7—C8	123.4 (6)
C2—C1—C10	122.6 (5)	C7—C8—C9	119.9 (7)
C6—C1—C10	118.7 (5)	C8—C9—C10	120.6 (7)
O1—C2—C1	114.8 (5)	C1—C10—C9	120.8 (5)
O1—C2—C3	124.3 (5)	C3—C4—H4	121.00
C1—C2—C3	120.9 (5)	C5—C4—H4	120.00
Br1—C3—C4	117.9 (5)	C6—C7—H7	118.00
C2—C3—C4	121.7 (6)	C8—C7—H7	118.00
Br1—C3—C2	120.4 (5)	C7—C8—H8	120.00
C3—C4—C5	119.1 (6)	C9—C8—H8	120.00
Br2—C5—C6	119.2 (4)	C8—C9—H9	120.00
C4—C5—C6	122.9 (6)	C10—C9—H9	120.00
Br2—C5—C4	117.8 (5)	C1—C10—H10	120.00
C1—C6—C7	116.6 (5)	C9—C10—H10	120.00
C5—C6—C7	126.9 (6)		
C6—C1—C2—O1	179.3 (5)	Br1—C3—C4—C5	-178.8 (4)
C6—C1—C2—C3	-1.5 (8)	C2—C3—C4—C5	-0.9 (9)
C10—C1—C2—O1	1.0 (8)	C3—C4—C5—Br2	176.9 (4)
C10—C1—C2—C3	-179.8 (6)	C3—C4—C5—C6	0.5 (9)
C2—C1—C6—C5	1.0 (8)	Br2—C5—C6—C1	-176.9 (4)
C2—C1—C6—C7	-179.7 (5)	Br2—C5—C6—C7	3.8 (9)
C10—C1—C6—C5	179.4 (5)	C4—C5—C6—C1	-0.5 (9)

C10—C1—C6—C7	-1.3 (8)	C4—C5—C6—C7	-179.8 (6)
C2—C1—C10—C9	179.2 (6)	C1—C6—C7—C8	0.4 (9)
C6—C1—C10—C9	0.9 (9)	C5—C6—C7—C8	179.6 (6)
O1—C2—C3—Br1	-1.5 (8)	C6—C7—C8—C9	1.0 (10)
O1—C2—C3—C4	-179.4 (5)	C7—C8—C9—C10	-1.5 (10)
C1—C2—C3—Br1	179.3 (4)	C8—C9—C10—C1	0.5 (10)
C1—C2—C3—C4	1.4 (9)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1 $\cdots$ Br1	0.82	2.60	3.107 (5)	122
O1—H1 $\cdots$ O1 <sup>i</sup>	0.82	2.21	2.893 (6)	141

Symmetry codes: (i)  $x-1/2, -y+1/2, -z$ .

Fig. 1

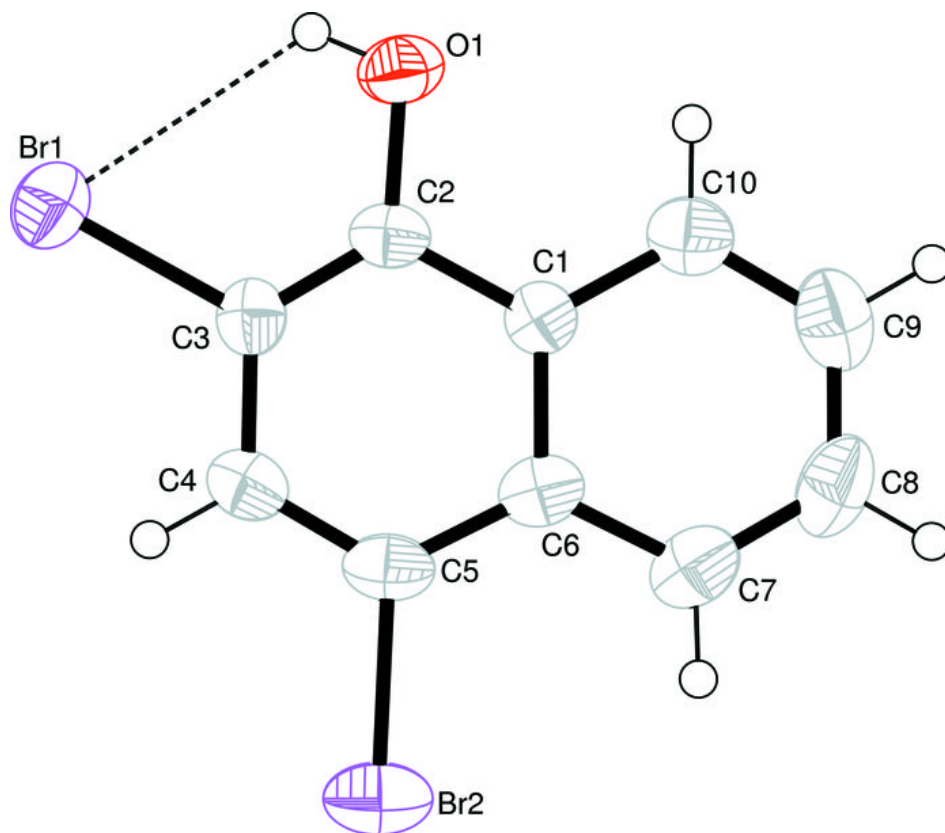




Fig. 2

